The Best Solvent And Extraction Time In Pectin Production made From Waste Of Jackfruit (Bark and Straw)

Wignyanto*, Nur Lailatul Rahmah, and Athika Dyah Margani
Department of Agro Industrial Technology,Brawijaya University
Jl. Veteran-Malang 65145, Indonesia
e-mail : wignyanto@ub.ac.id*

Abstract

Jackfruit barks are known to have fairly high pectin content, by 4.7% on wet and 22.5% on dry barks. The content is sufficiently highboy make it worthy of being raw material for producing pectin. Pectin is a polygalacturonic acid containing methyl ester. Pectin is a high-value functional food that is widely used as gelling agent and stabilizer. Approximately 65 until 80% jackfruit barks were wasted from the industry and it were only useful for animal feed . To increase the added value of it, so that jackfruit barks were extracted to get the pectin. This research used randomized block design (RBD) with 2 factors:(i)the type of solvents(hydrochloric and citric acids, with pH = 1.5) and (ii) extraction times (30, 90 and 150 minutes). Each factor is conducted in triplicate in order to get 18 units of experiment. Before analyzed, the jackfruit waste (bark and straw) was powdered during sample preparation. Later on, the yield, equivalent weight, methoxyl, galacturonic acid, and ash contents of the powdered, then were analyzed and used as pectin's parameters. Multiple attribute method (Zeleny method) was used to determine which combination of treatments that produced pectin with the best pectin's parameter. The best pectin made from jackfruit waste is derived from the combination of citric acid solvent and the extraction time of 150 minutes, resulting in yield of 10.21%, methoxyl content of 9.25%, equivalent weight of 1088.43 g/mol, ash content of 2.79%, and galacturonic acid content of 47.40%.

Keywords: *Extraction time, jackfruit waste, pectin, solvent*

1. INTRODUCTION

Jackfruit has been widely processed into chips, fruit juice, lunk head, sweets, syrup, jam, and pasta, where the waste produced can reach up to 65-80% of the total weight of the fruit.The waste consists of bark, seed and straw. The straw has a sizable portion, ranging from 40 to 50% of the total waste produced (Sugiarti, 2003).

Pectin is a polygalacturonic acid containing methyl ester. In addition, pectin is also a high-value functional food widely used as a gelling agent and stabilizer in fruit juice products, a jelly-making materiall (Willat *et al.*, 2006). In food and pharmaceutical industry, pectin is used as an emulsifier and stabilizer in food products and as an ingredient in drugs and cosmetics.

Recently, Indonesia's demand of pectin is fulfilled from abroad and has been increasing every year caused by insufficient supply from domestic pectin industry. In 1998, Indonesia imported 1,095 tons pectin worth IDR 5.2 million and the value has increased by 27% per year. However, there has been no previous research on the influence of solvent type and the extraction time on the properties of pectin made from jackfruit waste at the same pH condition. According to Garna *et al*. (2007), acid type can affect the properties of the resulting pectin. The use of different acids associated with their different hydrolysis levels due to differences in their concentration and

strength of the acid will produce pectin with different properties.

According to Rachmawan (2005), extraction time influences contact or diffusion between extractant and raw materials. The better the diffusion occurred, the higher the pectin obtained. Therefore, it is necessary to study the influence of solvent types (hydrochloric and citric acids) and extraction time on the properties of pectin made from jackfruit waste. The result is expected to be an early initiation step to develop pectin industry in Indonesia.

2. MATERIALS AND METHODS

The main materials used in this study were bark and straw of jackfruit. Chemicals used for the extraction of pectin were distilled water, technical hydrochloric acid, technical citric acid, and technical alcohol.

The tools used wereknives, blender (National), hot plate (HP 220), stirrer, filter cloth, oven (Memmert), sieve, analytical balance (AND GR-200), pH meters (Ezodo), and titration apparatus.

A randomized block design (RBD) with two factors is used as experimental design of the study. The first factor consists of two levels, namely type of solvents (hydrochloric and citric acids, with pH = 1.5), while the second factor consists of three levels, namely extraction times (30, 90 and 150 minutes). Each treatment was conducted in triplicate in order to get 18 units of experiment.

Analysis was conducted on the yield, equivalent weight, methoxyl content, galacturonic acid content, ash content and total pectin (best results). The obtained data is analyzed by analysis of variance (ANOVA), and further tested using Least Significant Difference (LSD) test with a confidence interval of 5%. To determine which treatment combinations produced the best pectin, multiple methods of attributes were used (Zeleny, 1982). The procedures of the research are as follow:

1. Production of jackfruit waste flour
2. The flour was weighed, and then added with a solution of hydrochloric acid or citric acid with pH 1.5. Acid solution was prepared at pH 1.5. Ratio of flour to the solution was 1: 10. After it was heated with a hot plate stirrer at 85^{o}C for extraction time of 30, 90 and 150 minutes, then the extract was filtered using a filter cloth. The filtrate obtained was pectin filtrate.
3. Pectin filtrate was then heated at 90^{o}C for 2 hours, and cooled to room temperature.
4. Alcohol 96%was added to the filtrate with a ratio of filtrate to alcohol 1:1, and then precipitated for 1 hour. The mixture was then filtered using a filter cloth to obtain a precipitate of pectin
5. Precipitate was purified into two stages, first with 70% alcohol with ratio of precipitates to alcohol 1:1 and second with 96% alcohol with ratio of precipitates to alcohol 1:1.
6. Clump of pectin was dried at 55^{o}C for 12 hours.
7. Rough Pectin was obtained.
8. The yield, equivalent weight, methoxyl content, galacturonic acid content, ash content and total pectin of the obtained pectin were then analyzed.

3. RESULTS AND DISCUSSION

The results show that the use of citric and hydrochloric acid solvents at pH 1.5 resulted in pectin with the average yield value ranged from 4.3% to 10.21% (Figure 1).

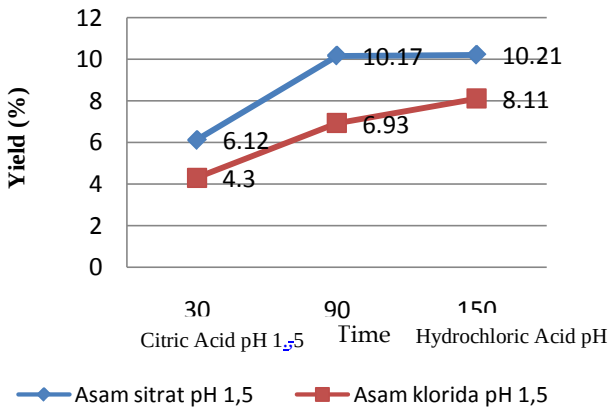

Figure1. Equivalent Yield of Pectin Flour Extracted with Different Solvent at Different Extraction Time.

Table 1. Effect of Extraction Time to theYield of Pectin made from Jackfruit Waste

Extraction Time	Mean ofyield (%)	LSD
30 minutes	5.21[a]	
90 minutes	8.54[a]	1,13
150 minutes	9.16[b]	

Description: numbers followed by the same letter are not significantly different at 0.05 levels

Table 2. Effect of Acid Solvent to the Mean Yield of Pectin made from Jackfruit Waste

Acid	Mean of Yield (%)	LSD
Hydrochloric acid	9.67[a]	1,13
Citric acid	13.25[b]	

Description: numbers followed by the same letter are not significantly different at 0.05 levels

Equivalent Weight

Equivalent weight is the amount of free galacturonic acid group content(non-esterified) in the pectin molecule chains (Meilina and Illah, 2012).In this study, the equivalent weight of pectin made from jackfruit waste, ranged from1088.43 to11574.08 g/ mol. Relationship between equivalent weight and extraction time of two types of acid solvent used for the extraction is shown in Figure 2.

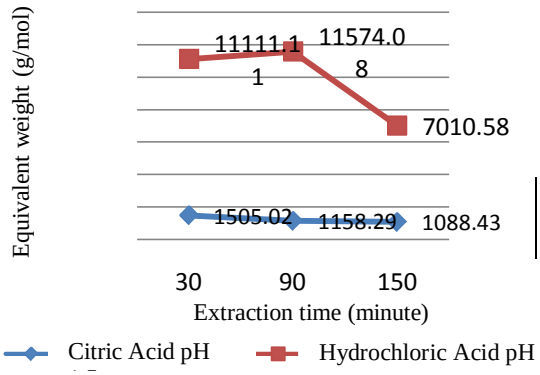

Figure2. Equivalent Weight of Pectin Flour Extracted with Different Solvent at Different Extraction Time.

Figure 2 showed that the pectin produced from material extracted with citric acid for 30 minutes had weight equivalent of 1505.02 g/mol, and was getting decreased to 1088.43 g/mol after 150 minutes extraction.

The decrease is caused by de-polymerization of pectin into pectic acid. According Meilina and Illah (2012), the longer the extraction time, the more pectin is depolymerized into pectic acid, resulting in more galacturonic acid groups that are not esterified. The result showed that longer extraction time tended to decrease equivalent weight of the pectin produced. Putra (2009) stated that the declination of equivalent weight is caused by pectin degradation and an increase in de-methylated pectin methoxyl groups.

The ANOVA tests showed that the type of solvent had a significant effect on the equivalent weight of pectin, whereas the extraction time had not, as well as the interaction. Effect of solvent type on the equivalent weight of the pectin produced is shown in Table 3.

Table 3. Effect of Acid Solvent Type to the Mean of Equivalent Weight of Pectin made from Jackfruit Waste.

Acid Solvent Type	Mean of Equivalent Weight(g/mol)	LSD
Hydrochloric acid	14847.88[a]	4215.73
Citric acid	1625.04[b]	

Description: numbers followed by the same letter are not significantly different at 0.05 levels

Methoxyl Content

Methoxyl content indicates the number of methyl ester groups in the pectin molecule (Putra, 2010).The number of methoxyl content is one of the important properties that influence the formation of pectin gels. Relationship between the two treatments, solvent type and length of extraction, on the methoxyl content is shown in Figure 3.

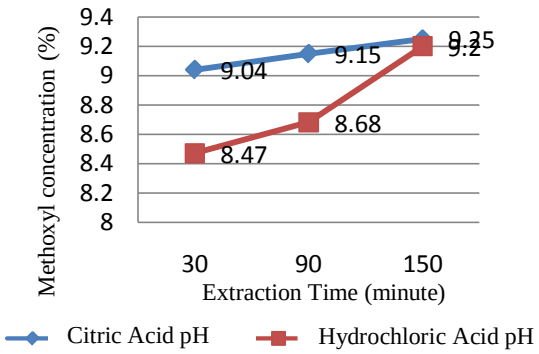

Figure 3. Methoxyl Content in Pectin Flour made from Jackfruit Waste.

Based on Figure 3, it was known that on any combination of solvent type and extraction time, the content of methoxyl produced was similar, whereas the yield of pectin was not (Figure 1).This is presumably due to the similar capacity of the methoxyl content of the extracted pectin, whereas the pectin content varies in each treatment depend on treatment effect. Pectin contains galacturonic acid, acetic acid, L-arabinose, D-xylose, D-glucose, D-mannose, L-fructose and other compounds.

Based on the ANOVA result, types of solvent, extraction times, and their interaction did not significantly affect the methoxyl content of the pectin. This is due to the methoxyl content of each treatment was not significant because of the extracted methoxyl capacity was not different for all treatments.

Ash Content

Effect of solvent types and time of extraction on ash content of pectin made from jackfruit waste is shown in Figure 4.

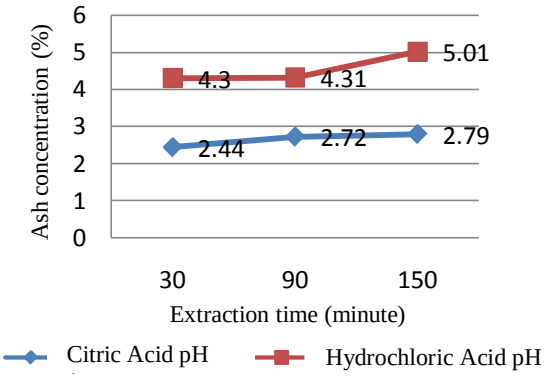

Figure 4. Mean of Ash Content in Pectin Flour made from Jackfruit Waste.

Based on Figure 4, it can be seen that the ash content of pectin extracted with hydrochloric acid produced a higher ash content, ranged from4.3to 5.01%, whereas pectin extracted with citric acid resulted in a lower ash content, ranged from2.44to 2.79%.

Purwoko (2011) mentioned that the use of chemicals such as hydrochloric acid or alcohol containing impurities in a certain amount is presumably to be the cause of the increased ash content of pectin.

Based on the analysis of variance, acid solvent and extraction time showed a significant effect on ash content. Furthermore, based on the result of LSD test, there were significant differences of ash content on each treatment as shown in Table 4 and 5.

Table 4. Effect of Extraction Time on Ash Content in Pectin Flour

Time (minutes)	Mean of Ash Content (%)	LSD
30minutes	3,37[a]	
90minutes	3,52[a]	0,44
150minutes	3,90[b]	

Description: numbers followed by the same letter are not significantly different at 0.05 levels

Table 5. The Effect of Acid Solvent Types on Ash Content in Pectin Flour

Acid	Mean of Ash content (%)	LSD
Citric acid	3.97[a]	
Hydrochloric acid	6.81[b]	0,44

Description: numbers followed by the same letter are not significantly different at 0.05levels

Galacturonic acid content

In this study, the content of galacturonic acid in pectin ranged from 21.35to 47.40% as shown in Figure 5.

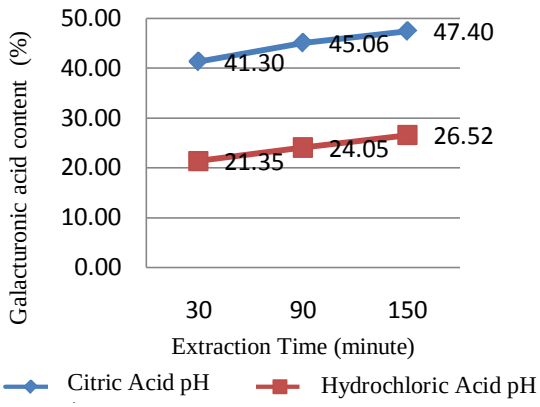

Figure 5. Galacturonic acid content in pectin flour made from jackfruit waste.

Based on Figure 5 it can be seen that the content of galacturonic extracted with citric acid solvents was higher than that extracted with hydrochloric acid. The content of Galacturonic acid extracted with citric acid ranged from 41.30 to 47.40%, while that extracted with hydrochloric acid ranged from 21.35 to 26.52%. The lower content of galacturonic acid extracted with hydrochloric acid is presumably because this strong acid increases the decarboxylation process (a chemical process that leadsto a carboxyl group apart from its compound) on pectin compounds, which lead to the decomposition and reduction of galacturonic acid.

The content of galacturonic acid extracted with hydrochloric acid was less than 35% and did not meet the Codex requirement (1972). This is presumably due to differences in the composition of the compounds contained in the pectin. These compounds affect the content of the produced galacturonic acid as shown in Table 6.

Based on the analysis of variance, the acid solvent types had a significant influence on the content of galacturonic acid, whereas the extraction time had not.

Effect of solvent on the content of produced galacturonic acid is shown in Table 6.

Table 6. The Effect of Acid Solvent Types on Galacturonic Acid Content in Pectin Flour.

Acid	Mean of galacturonic acid(%)	LSD
Hydrochloric acid	35.96[a]	9,04
Citric acid	66.88[b]	

Description: numbers followed by the same letter are not significantly different at 0.05 levels

Best Treatment

Pectin with best selected parameters was obtained from the combination of citric acid solvent and extraction time of 150 minutes as shown in Table 7. Based on Table 7, it can be seen that the current results have met the standards, especially for the methoxyl and galacturonic acid contents. When compared with the previous study, the content of our pectin's yield and methoxyl is higher. However, the ash content and

equivalent weight of the current result are lower. The pectin, which was produced from the waste of jackfruit, is categorized as pectin with high methoxyl content (>7%).

Table7. Pectin's parameters extracted with citric acid for 150 minutes

Parameter	Result of current study[a]	Result of previous study[b] (Putra, 2010)	Criteria
Yield	10.21 %	4.54 %	-
Equivalent Weight	1088.43 g/mol	3022.24 g/mol	-
Methoxyl content	9.25 %	8.16 %	High Methoxyl content ≥7% Low Methoxyl content ≤7%
Ash content	2.79 %	2.82 %	≥1 %
Galacturonic acid content	47.40 %	88.01%	≥ 35 %
Total Pectin	33.46 %	-	-

Description: [a]wet raw material (85.92% of moisture content), [b]dried raw material (14.00% of moisture content)

Total pectin was determined later on the sample with best parameter values to find out how much pectin contained in the extracted pectin yield. The higher the total pectin is contained, the better and the purer the pectin is. The low number of total pectin shows impurities that have been extracted and coagulated during the extraction process (Rachmawan *et al.*, 2005).

Total pectin obtained from the sample that extracted with citric acid solvent was33.46%, much more higher than total pectin extracted from cocoa rind in previous study (ranged from 14.27 to26.73%) conducted by Rachmawan *et al.* (2005) In the current study, total pectin contained in wet jackfruit waste (bark and straw) was 1.51%, while the total pectin contained in jackfruit waste flour was 11.2%. The flour waste has been dried first. According to Fitriani (2003), drying is a dehydration process which reduces the moisture content of the material. The reduction can increase the proportion of total pectin to total material. Thus, the lower the moisture content, the higher the content of total pectin obtained.

Extracted total pectin from the research was 33.46%. The low content of total pectin obtained in this study is presumably caused by unfavorable process i.e. alcohol leaching, and the presence of extracted and coagulated impurities during the extraction process.

According to Fitriani (2003), the purity of pectin is mostly influenced by acid leaching by alcohol. If the leaching does not eliminate the acid, pectin content will be low. It was believed that the obtained pectin flour was still containing ingredients other than pectin, which might be entrained during extraction and coagulation process.

Citric acid solvents produce good characteristics because citric acid is a weak acid. Acid strength influences characteristics of pectin, caused by its influence on protopectin hydrolysis into pectin (Rachmawan *et al*, 2005). Long extraction also affects the characteristics of the pectin. The longer the extraction process, the more yield of pectin produced and the higher the content of methoxyl. According to Budiyanto and Yulianingsih (2008), this can be due to the free carboxyl groups are esterified.

CONCLUSION

1. Pectin produced in this study has met the content standards of methoxyl and galacturonic acid. The methoxyl content ranged from 8.47 to 9.25% (including high methoxyl content), while the content of galacturonic acid was at least 35%.
2. The best pectin, made from jackfruit waste,is derived from the treatment with citric acid solvent and 150 minutes extraction time. The results has a yield value of 10.21%, methoxyl content of 9.25%, equivalent weight of 1088.43 g / mol, ash content of 2.79%, and 47.40% galacturonic acid levels.

RECOMMENDATION/ FUTURE WORK

Further research on the factors that influence the content of ash in pectin is required in order to meet its recommended maximum value.

REFERENCES

Budiyanto, A. dan Yulianingsih. 2008. Pengaruh Suhu dan Waktu Ekstraksi terhadap Karakter Pektin dari Ampas Jeruk Siam *(Citrus nobilis L). J. Pascapanen* 5 (2) : 37-44.

Fitriani, V. 2003. Ekstraksi dan Karakteristik Pektin dari Kulit Jeruk Lemon *(Citrus medica* var *Lemon)*. *Skripsi*. Fakultas Teknologi Pertanian. Institusi Pertanian Bogor. Bogor

Garna, H., Nicholasmabon C., Robert C., Cornet K., Nott Herv'ELegros, Bernardwathelet and A. Paquot. 2007. Effect of Extraction Conditions on the Yield and Purity of Apple Pomance Pectin Precipitated but Not Washed by Alcohol. *J. Food Science*. 72 (1).

Meilina, H. danIllah S. 2012. Produksi Pektin dari Kulit Jeruk Lemon. *Prosiding Simposium Nasional Polimer V*. IPB. Bogor. Hal 117-126.

Purwoko. 2011. Pembuatan Pektin dari Buah Pepaya (Carica papaya L) Sisa Sadap. *J. Teknik industry pertanian* 12 (1): 8-13.

Putra, I. N. K., Komang, A. N., dan Agus S. D. 2009. Optimasi Suhu dan Waktu pada Proses Ekstraksi Pektin Ampas Buah Nangka. *Prosiding Seminar Nasional* FTP UNUD, Denpasar, hal. 1-4.

Putra, I. N. K. 2010. Optimasi Proses Ekstraksi Pektin Dami Buah Nangka *(Artocarpus heterphyllus* Lamk). *J.Agritech* 30 (3): 1-14.

Rachmawan, A., Lestari D., Dwierra E., dan Djoko S. 2005. Ekstraksi dan Karakteristik Pektin dari Kulit Buah Kakao. *J. Ilmiah Pertanian Gakuryoku* 11(2): 190-194.

Sugiarti . 2003. *Pengaruh Asam Sitrat dan Gula Terhadap Mutu Selai dari Dami Nangka Varietas Nangka Kunir (Artocarpus heterophyllus)*. Dilihat 20 Maret 2012. (http://digilib.itb.ac.id/gdl.php?mod=browse&op=read&id=jiptumm-gdl-s1-2003-sugiarti98-960).

Willat, W.G.T., P. Knox and Mikkelsen. 2006. Pectin :New Insights Into on Old Polymer are Starting to Gel. *J. Trends in Food Science and Technology* 17: 97–104.